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05SEP02 E746021-1 010136
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# Request for grant of a patent

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| | | |
|---|---|---|
| 1. | Your reference | GB 21.1105 |
| 2. | Patent application number *(The Patent Office will fill in this part)* | 0220604.3    5 SEP 2002 |
| 3. | Full name, address and postcode of the or of each applicant *(underline all surnames)* | SCHLUMBERGER HOLDINGS LIMITED<br><br>PO BOX 71<br>Craigmuir Chambers<br>Road Town - Tortola |
| | Patents ADP number *(if you know it)* | 7236326001 |
| | If the applicant is a corporate body, give the country/state of its incorporation | British Virgin Islands |
| 4. | Title of the invention | WELL TREATMENT FLUIDS CONTAINING FIBERS |
| 5. | Name of your agent *(if you have one)*<br><br>"Address for service" in the United Kingdom to which all correspondence should be sent *(including the postcode)* | Brian STOOLE<br>SENSA, Gamma House, Enterprise Road<br>Chilworth Science Park, Southampton<br>Hampshire SO16 7NS,<br>England |
| | Patents ADP number *(if you know it)* | 8021560002 |

| | | Country | Priority application number *(if you know it)* | Date of filing *(day / month / year)* |
|---|---|---|---|---|
| 6. | If you are declaring priority from one or more earlier patent applications, give the country and the date of filing of the or of each of these earlier applications and *(if you know it)* the or each application number | | | |

| | | Number of earlier application | Date of filing *(day / month / year)* |
|---|---|---|---|
| 7. | If this application is divided or otherwise derived from an earlier UK application, give the number and the filing date of the earlier application | | |

| | | |
|---|---|---|
| 8. | Is a statement of inventorship and of right to grant of a patent required in support of this request? *(Answer 'Yes' if:*<br>*a) any applicant named in part 3 is not an inventor, or*<br>*b) there is an inventor who is not named as an applicant, or*<br>*c) any named applicant is a corporate body.*<br>*See note (d))* | YES |

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| Continuation sheets of this form | – |
| Description | 8 |
| Claim *(s)* | 2 |
| Abstract | 1 |
| Drawing *(s)* | 4 +4 |

10. If you are also filing any of the following, state how many against each item.

| | |
|---|---|
| Priority documents | 0 |
| Translations of priority documents | 0 |
| Statement of inventorship and right to grant of a patent *(Patents Form 7/77)* | 1 |
| Request for preliminary examination and search *(Patents Form 9/77)* | 1 |
| Request for substantive examination *(Patents Form 10/77)* | 1 |
| Any other documents *(please specify)* | Fee Sheet, Cover Letter |

11. I/We request the grant of a patent on the basis of this application.

Signature

Martin HYDEN Chartered Patent Agent

Date September 3rd, 02

12. Name and daytime telephone number of person to contact in the United Kingdom

Brian STOOLE
02380 765573

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# WELL TREATMENT FLUIDS CONTAINING FIBERS

The present invention relates to well treatment fluids containing particles and fibres. In particular, it relates to such fluids in which the sedimentation of the particles or fibres in the fluid is prevented or hindered. Such fluids find applications in well cementing and stimulation operations.

The use of particles or fibres in well treatment fluids such as cements has been previously proposed. One such example is described in European Patent No. 1086057 which describes the use of amorphous cast-iron platelet particles in oil-well cements to provide added toughness and impact resistance. However, there is a large density difference between the particle and the base fluid – the cement slurry with which the particles are mixed – so special care must be taken to prevent the sedimentation of the particles. When the particles are metallic, for instance based on cast iron, the density difference is commonly around 5000 kg/m$^3$. Preventing the sedimentation of these fibres is usually ensured by viscosifying the base fluid. The rheology of the base fluid is characterized by a minimum of two parameters, the high shear rate viscosity and the yield stress, which quantifies the low shear rate viscosity of the fluid. There are many drawbacks to viscosifying the base fluid, for instance increase friction pressure drops when the fluid is pumped through tubulars, difficulties in mixing it and increased cost due to the use of viscosifying agents.

It is an object of the invention to provide a fibre-containing well treatment fluid in which the settling of particles or fibres is inhibited.

The present invention addresses the problem of fibre or particle settling by providing a fluid containing two fibre components - or a particle and a fibre - of differing properties.

In accordance with the present invention, there is provided a well treatment fluid, comprising a base fluid; a first fibre or particle component that it substantially more dense than the base fluid; and a second fibre component that has a density closer to that of the base fluid and is relatively flexible. While the term "first fibre component"

is used in this application, this component can also be present in a particulate forms and the scope of this term is to be interpreted accordingly without restricting the application of the invention to fibres only.

The second fibre component forms a network in the base fluid which traps the fibres of the first fibre component and prevents or hinders settling.

The first fibre component is typically a metallic material, such as amorphous cast iron, which can be present in the form of platelet-like structures having an average length that is less than 10mm. Such a fibre is relatively short, dense and rigid.

The second fibre component is typically a glass or polymeric material in the form of long, flexible fibres or ribbons. Such materials typically have a density close to that of the base fluid (cement or fracturing fluids) and a fibre length in the range 5 – 35mm.

The second fibre component is typically present in an amount of less than 10% by mass of the total amount of fibre in the fluid.

By adopting the approach of this invention, it is possible to maintain the fibres in suspension without the need to increase the viscosity of the fluid with the inherent operational problems that can be caused thereby.

The present invention will now be described by way of examples and with reference to the accompanying drawings, in which:
Figure 1 shows the experimental apparatus;
Figure 2 show plots of pressure vs. time for runs A2 and A3;
Figures 3a and 3b show plots of sedimentation time vs. effective concentrations C1 and C2 respectively; and
Figures 4a and 4b show photographs of sediment cake with Samples 1 and 5 respectively.

Sedimentation experiments are performed in a vertical tube T, with a diameter of 5 cm and a length of 80 cm. The fibre concentration is determined by monitoring the

pressure gradient using a Validyne differential pressure gauges ΔP1, as shown on Fig. 1. The differential pressure gauges ΔP1, is calibrated using a column of water (). All measurements are expressed in terms of density, by converting the pressure records according to the following calibration data.

| Transducer | ΔP1 | ΔP2 | ΔP3 |
|---|---|---|---|
| Membrane nb and range | 30 – 86 mbar (1.25 psi) | 26 – 35 mbar (0.5 psi) | 22 – 14 mbar (0.2 psi) |
| Sensitivity / calibration slope | 2.0 V / mbar | 3.76 V / mbar | 3.69 V / mbar |

$\Delta\rho_i = \Delta P_i / g\Delta H_i$ Eq. 1

The experimental procedure used comprises the following steps:
The sedimentation tube is filled with water and all tubes are purged from air bubbles. The equilibrium transducer output is recorded to be used as baseline.

The base fluid is mixed: 12 g of biozan (a biopolymer) are added to 3 L of water and the solution is stirred for 40 min to allow full hydration of the polymer, using a paddle mixer. Antifoam and biocide are added. This base fluid is chosen for its rheology and essentially inert behaviour towards the fibres used in the experiments while corresponding closely to the behaviour of cementing and fracturing fluids.

The base fluid is tested to determine its rheology.

The dense fibres (SG Seva Fibraflex FF5E0 "Fibraflex") are added to the base fluid. When flexible fibres are used, 500 mL of base fluid are poured in a waring blender. The flexible fibres are dispersed by rotating at low speed. The rotation speed is adjusted in order to keep the vortex. The suspension is then poured back in the main container where Fibraflex fibres are added.

In the experiments, the suspension is either poured into the sedimentation tube or is pumped using a peristaltic pump to fill the tube from the bottom.

The pressure gradients are recorded on paper for a period of time ranging from 1 hour to overnight. The pressure decay is fitted with an exponential function allowing determination of the time constant.

Measurements are interpreted in terms of excess density with respect to the base fluid. Knowing the density and concentration of FIBRAFLEX fibres (100 g/L), the theoretical density of the homogeneous suspension is 1086 g/L. The excess density is therefore about 86 g/L.

The rheology of five batches of base fluid is monitored using a Fann 35, R1B1F1, to assess its reproducibility. The corresponding data and plot are shown below (Table 1). The rheology of the polymer solution is stable for more than one day.

| Sample | A | B | C | D | E |
|---|---|---|---|---|---|
| 300/200/100/60/30/6/3 RPM Gel: 10 min / 1 s / stir readings | 25/21/17/15 /12/8/7 12/9/8 | 27/23/19/17/ 13/9/8 14/10/9 | 27/22/18/15/ 13/9/8 12/9/8 | 27/23/19/17/ 14/10/9 12/10/9 | 26/22/18/15/ 13/9/8 - |
| HB parameters: K (Pa.s$^{n}$) / n / $T_0$ (Pa) | 0.67 / 0.437 / 1.94 | 0.97 / 0.392 / 2.0 | 0.346 / 0.54 / 3.1 | 0.82 / 0.401 / 3.05 | 0.583 / 0.45 / 2.94 |
| Bingham parameters PV (cP) / YP (Pa) | 13.3 / 5.8 | 12.4 / 7.03 | 15.3 / 5.75 | 13.3 / 6.75 | 14.3 / 5.85 |

Table 1

Six different flexible fibres (Samples 1 – 6) are studied. Their properties are gathered in Table 2 below.

Sample 1: short polyamide (nylon 6-6) fibres ().

Sample 2: long polyamide fibres,.

Sample 3: polypropylene ribbons, with a broad length distribution.

Sample 4: glass fibres with 40% water.

Sample 5: novoloid fibres with 20% water.

Sample 6: PET fibre. Its formulation includes a dispersant to enhance its dispersability in water. When the fibres are cleaned with an organic solvent, they become very difficult to disperse in water.

| Sample | FIBRAFLEX | 1 | 2 | 5 | 3 | 4 | 6 |
|---|---|---|---|---|---|---|---|

| Material | Cast iron | polyamide | polyamide | novoloid | polypropylene | fiberglass | Polyester |
|---|---|---|---|---|---|---|---|
| Shape | platelets | rods | rods | rods | platelets | rods | rods |
| Length | 5 mm | 12 mm | 19 mm | 20 mm | 12 - 35 mm | 12 mm | 6 mm |
| Diameter or width | 0.8 mm wide | 18 µm | 18 µm | 21 µm | 0.8 mm wide | 20 µm | ~ 10 µm |

Table 2

The following suspension compositions are analysed, where the concentrations are defined per litre of water.

| Reference and placement method | Base fluid | Fibres | Sedimentation time (minutes), mean fluctuation (%) |
|---|---|---|---|
| A1<br>Poured from top | Biozan 4g/L<br>Antifoam 1g/L | Fibraflex 100g/L<br>Sample 2 2g/L | 154 (0.31%) |
| A2<br>Poured from top | Biozan 4g/L<br>Antifoam 1g/L<br>Biocide 1g/L | Fibraflex 100g/L | 4 (1.2%) |
| A3<br>Poured from top | Biozan 4g/L<br>Antifoam 1g/L<br>Biocide 1g/L | Fibraflex 100 g/L<br>Sample 1 2g/L | 101 (0.35%) |
| A4<br>Poured from top | Biozan 4g/L<br>Antifoam 1g/L<br>Biocide 1g/L | Fibraflex 100 g/L<br>Sample 1: 3g/L | 134 (0.35%) |
| A5<br>Pumped from bottom | Biozan 4g/L<br>Antifoam 1g/L<br>Biocide 1g/L | Fibraflex 100 g/L<br>Sample 1: 4g/L | 544 (0.18%) |
| A6<br>Pumped from bottom | Biozan 4g/L<br>Antifoam 1g/L<br>Biocide 1g/L | Fibraflex 100 g/L<br>Sample 1: 1g/L | 52 (0.38%) |
| A7<br>Pumped from bottom | Biozan 4g/L<br>Antifoam 1g/L<br>Biocide 1g/L | Fibraflex 100 g/L<br>Sample 5: 2g/L | 571 (0.34%) |
| A8<br>Pumped from bottom | Biozan 4g/L<br>Antifoam 1g/L<br>Biocide 1g/L | Fibraflex 100 g/L<br>Sample 4: 2g/L | 65 (0.21%) |
| A9<br>Pumped from bottom | Biozan 4g/L<br>Antifoam 1g/L<br>Biocide 1g/L | Fibraflex 100 g/L<br>Sample 3: 2g/L | 47 (0.62%) |
| A10 | Biozan 4g/L | Fibraflex 100 g/L | 100 (0.13%) |

| | | | |
|---|---|---|---|
| Pumped from bottom | Antifoam 1g/L<br>Biocide 1g/L | Sample 5: 1 g/L | |
| A11<br>Pumped from bottom | Biozan 4g/L<br>Antifoam 1g/L<br>Biocide 1g/L | Fibraflex 100 g/L<br>Sample 6: 2 g/L | 2100 (0.11%) |
| A12<br>Pumped from bottom | Biozan 4g/L<br>Antifoam 1g/L<br>Biocide 1g/L | Fibraflex 100 g/L<br>Sample 6: 1 g/L | 283 (0.15%) |

Table 3

A first general observation is that the initial excess density (Figure 2) is in quite good agreement with the theoretical value of 86 g/L. Another general observation is that large pressure fluctuations are observed for all experiments, starting at the beginning and lasting until no more FIBRAFLEX fibres are left in suspension in between the pressure ports of the transducer. From visual observations, two phenomena may explain these observations:

The FIBRAFLEX fibres tend to settle as large aggregates.

In a few cases, convection cells are clearly observed especially when the settling velocity is fast.

A measure of the amplitude of these fluctuations is provided (Table 3) as the mean square difference between the measurements and the exponential fit.

Results for runs A2 and A3 are shown in Fig. 2 and are typical.
The sedimentation times are plotted versus the concentration of flexible fibres on Figs. 3a and 3b. Logically, the sedimentation time increases – faster than linearly – when the concentration of flexible fibres increases. Also, the more dispersed the fibres are, the strongest the effect: Sample 6 are dispersed in individual fibres, Sample 5 are slightly more difficult to disperse while the polyamide and polypropylene fibres are clearly hydrophobic and remain stuck together. In summary, the experimental data fall in three groups:
Very large effect, Sample 6;
Average effect: Sample 5, Sample 4, Sample 2, Sample 1;

Small effect: Sample 3.

In dilute suspensions, the relevant parameters that characterize the settling of fibres are their hydrodynamic size and their effective concentration calculated based on this size. If N is the number of fibres per unit volume and L their length and d the fibre diameter, the relevant dimensionless numbers are:

$$
\begin{aligned}
c_1 &= N(L/2)^3 \\
c_2 &= N(L/2)^2(d/2) \quad \text{Eq. 2} \\
c_3 &= 2\pi N(L/2)(d/2)^2
\end{aligned}
$$

The first number represents the hydrodynamic volume, in dilute conditions. The second number is the volume of the disks whose diameter is equal to the fibre length: it is considered to be the relevant parameter in semi-dilute regime. The third number is the material concentration.

For $c_1 < 1$, the regime is dilute; if $c_1 > 1 > c_2$ this is a semi-dilute regime and the suspension is considered to be concentrated when $c_2 > 1$. Table 4 provides these effective concentrations, based on a concentration of flexible fibres of 2 g/L.

| | Rods | | | | | Ribbons | |
|---|---|---|---|---|---|---|---|
| | Sample 4 | Sample 5 | Sample 2 | Sample 1 | Sample 6 | Sample 3 | Fibraflex |
| Cylinder diam. / Width (µm) | 20 | 21 | 18 | 18 | 10 | 800 | 1000 |
| Thickness (µm) | - | - | - | - | - | 23 | 25 |
| Length (mm) | 12 | 20 | 19 | 12 | 6 | 12 to 35 | 5 |
| Density (kg/L) | 2.5 | 1.27 | 1.1 | 1.1 | 1.37 | 0.9 | 7.2 |
| Concentration (g/L) | 2 | 2 | 2 | 2 | 2 | 2 | 100 |
| Nb/volume ($m^{-3}$) | 5.31E+07 | 5.68E+07 | 9.40E+07 | 1.49E+08 | 7.44E+08 | 1.01E+07 | 1.11E+08 |
| Material conc., $c_3$ (vol %) | 0.08% | 0.16% | 0.18% | 0.18% | 0.15% | 0.22% | 1.39% |
| Effective conc. $c_1$ / $c_2$ | 11 / 0.02 | 56 / 0.06 | 80 / 0.08 | 32 / 0.05 | 20 / 0.03 | 2 / 0.15 | 2 / 0.3 |

Table 4

Clearly, the suspensions must be considered to be in the semi-dilute regime.

The fact that the fibres are intimately interpenetrating ($c_1 >> 1$) explains the suspension properties: they form a kind of a gel that physically hinders the settling of the heavy FIBRAFLEX particles. It is possible to make sure that, when the

FIBRAFLEX particles settle, they do so through the network of flexible fibres. The clear supernatant liquid at the top of the column still contains flexible fibres and the sediment deposited at the bottom of the tube contains a mixture of both fibres, clearly visible in Figures 4a and 4b. When flexible fibres are present, the cake of FIBRAFLEX particles is much less compact to a point that sometimes it can be put back in flow with no external mixing.

Using its hydrodynamic volume and Stokes law, the settling velocity of a single FIBRAFLEX particle would be around 0.5 mm/s, considering an effective solution viscosity of 0.5 Pa.s (at 10 $s^{-1}$). This value is in agreement with the mean settling velocity of experiment A2 (no flexible fibres used), v ~ 20 cm / 4 mn = 0.8 mm/s.

The exponential behaviour of all measured data corresponds to a sedimentation regime governed by interactions between sedimenting particles: for non-interacting particles, a linear behaviour should be observed corresponding to the downward displacement of the upper front. This observation re-enforce the fact that the fibre suspension is in a semi-dilute regime. .

The density fluctuations observed in almost all experiments may originate from the difficulty in preparing homogeneous fibres suspensions, especially when the fibres are hydrophobic (Samples 2 and 3). Also, the significant length of the fibres can lead to wrapping around any rotating instrument, possibly resulting in kind of "balls of yarn". There is some correlation between the amplitude of these fluctuations and the nature of the fibres: the amplitude decreases for large fibre concentration and hydrophilically treated materials (Samples 4 and 5).

CLAIMS

1 A well treatment fluid, comprising:

i) a base fluid;

ii) a first fibre component that it substantially more dense than the base fluid; and

iii) a second fibre component that has a density close to that of the base fluid and is relatively flexible.

2 A well treatment fluid as claimed in claim 1, wherein the first fibre component comprises a metal.

3 A well treatment fluid as claimed in claim 2, wherein the metallic fibre comprises amorphous cast iron.

4 A well treatment fluid as claimed in claim 3, wherein the metallic fibres are flat, plate-like structures having an average length less than 10mm.

5 A well treatment fluid as claimed in any preceding claim, wherein the second fibre component comprises a glass, carbon or polymeric material.

6 A well treatment fluid as claimed in claim 5, wherein the fibres of the second fibre component are at least as long as, and thinner than, the first fibre component.

7 A well treatment fluid as claimed in claim 5, or 6, wherein the second fibre component has an average fibre length in the range 5 – 35 mm.

8 A well treatment fluid as claimed in claim 5, 6 or 7, wherein the second fibre component is present at a concentration of less than 10% by mass of the total fibre content of the fluid.

9 A well treatment fluid as claimed in any preceding claim, wherein the base fluid comprises a cement slurry.

10 A well treatment fluid as claimed in any of claims 1 – 8, wherein the base fluid comprises a fracturing fluid.

11 A well fluid as claimed in any preceding claim, wherein the base fluid exhibits shear-thinning behaviour.

12 A method of treating a well comprising pumping into the well a fluid comprising:

i) a base fluid;

ii) a first fibre component that it substantially more dense than the base fluid; and

iii) a second fibre component that has a density close to that of the base fluid and is relatively flexible.

13 A method as claimed in claim 12 comprising a cementing operation.

14 A method as claimed in claim 12, comprising a stimulation operation.

## ABSTRACT

The problem of fibre or particle settling in well treatment fluids is addressed by providing a fluid containing two fibre components of differing properties. Such a well treatment fluid, comprising a base fluid; a first fibre component that it substantially more dense than the base fluid; and a second fibre component that has a density close to that of the base fluid and is relatively flexible.

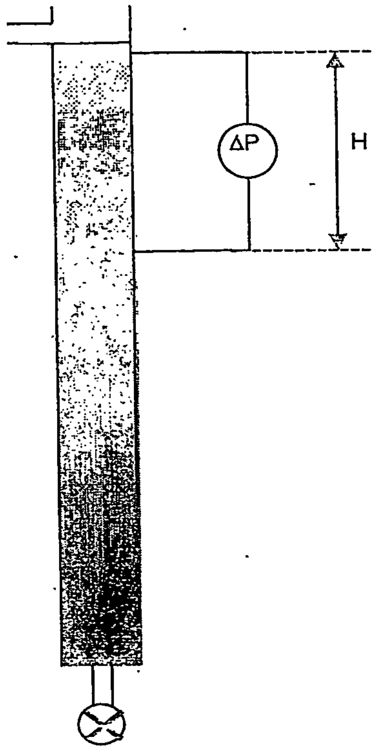
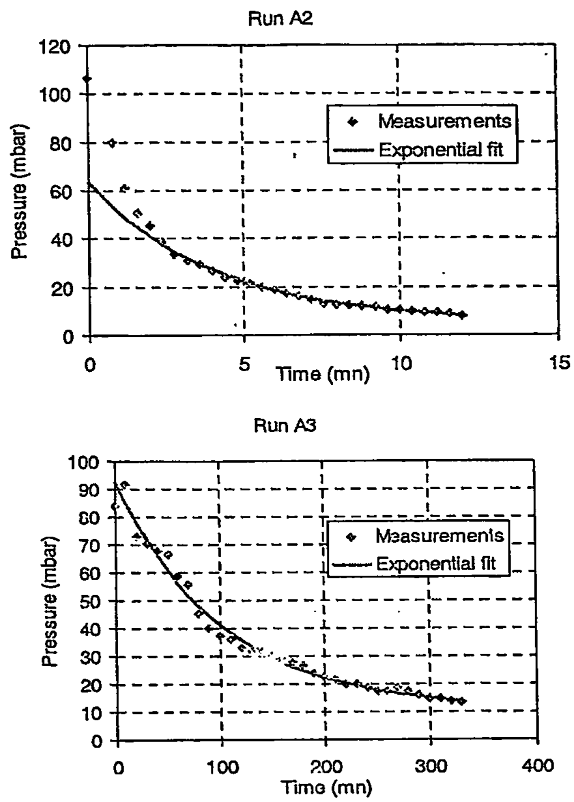



FIGURE 1


Run A2
120
100
80
60
40
20
0
Pressure (mbar)
Measurements
Exponential fit
0
5
10
15
Time (mn)
Run A3
100
90
80
70
60
50
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10
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Pressure (mbar)
Measurements
Exponential fit
0
100
200
300
400
Time (mn)

FIGURE 2

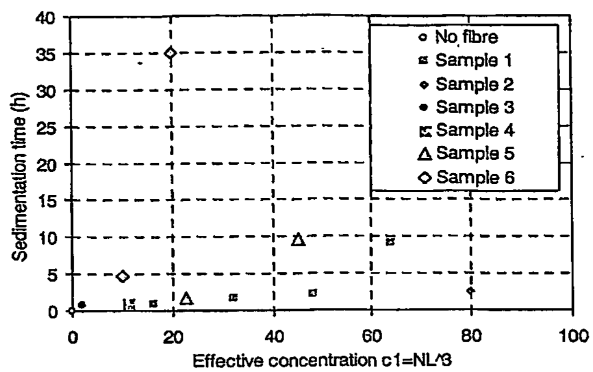


FIGURE 3A

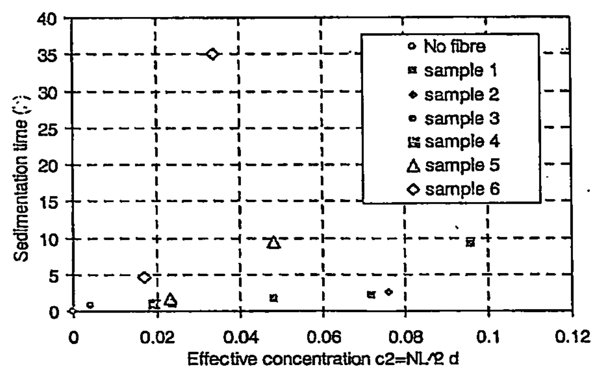


FIGURE 3B

FIGURE 4A

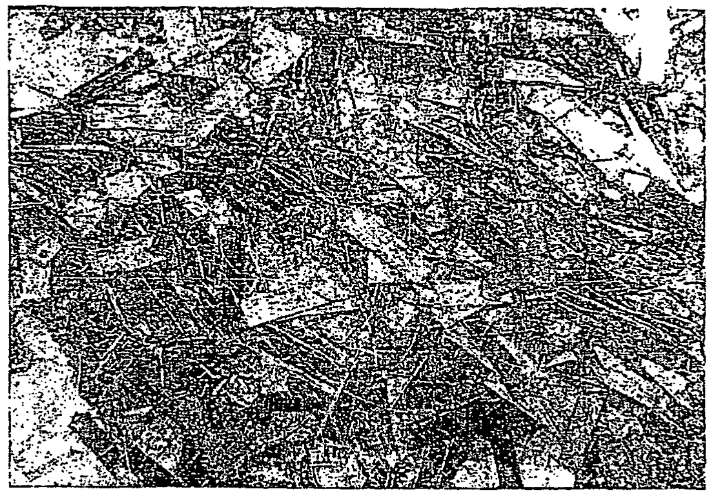

FIGURE 4B

PCT Application
EP0310001